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(71) **RECUPAC,
528 Avenue de Savoie, LECHEYLAS, XX (FR).**

(72) **TEDJAR, FAROUK (FR).
JUIF, FREDERIC (FR).**

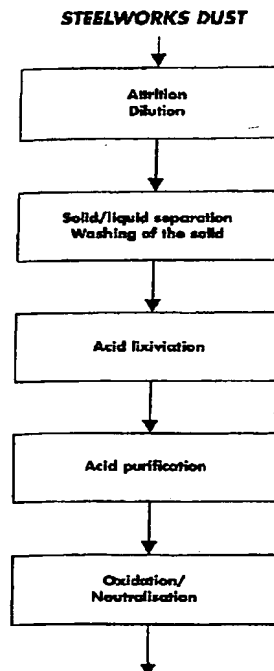
(74) **BERESKIN & PARR**

(54) **PROCEDE DE VALORISATION DES POUSSIERS D'ACIERIES**

(54) **METHOD FOR UPGRADING STEEL PLANT DUST**

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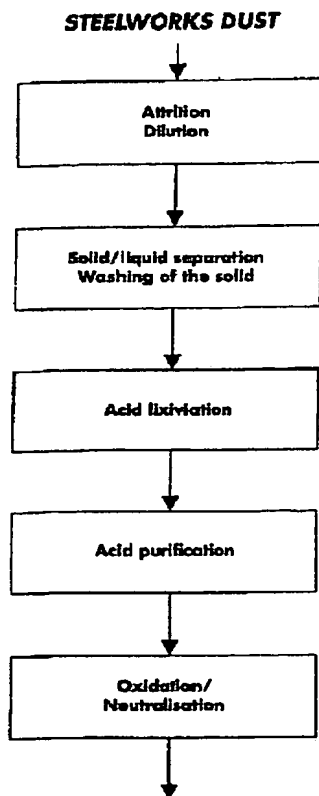
The invention concerns a method for upgrading steel plant dust to recuperate industrially reusable compounds. Said method comprises an attrition and dilution step, a solid/liquid separating and solid washing step, an acid leaching step, and acid purification step comprising a carburizing reaction or a carburizing and decalcification reaction. Said method is characterised in that it comprises an oxidation/neutralisation step which includes a step oxidizing iron into iron oxyhydroxides and/or iron oxides and a neutralizing reaction of the reaction medium.



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(71) Demandeur/Applicant:
RECUPAC, FR
(72) Inventeurs/Inventors:
TEDJAR, FAROUK, FR;
JUIF, FREDERIC, FR
(74) Agent: BERESKIN & PARR

(54) Titre : PROCEDE DE VALORISATION DES POUSSIÈRES D'ACIERIES
(54) Title: METHOD FOR UPGRADING STEEL PLANT DUST



(57) Abrégé/Abstract:

The invention concerns a method for upgrading steel plant dust to recuperate industrially reusable compounds. Said method comprises an attrition and dilution step, a solid/liquid separating and solid washing step, an acid leaching step, and acid

(57) Abrégé(suite)/Abstract(continued):

purification step comprising a carburizing reaction or a carburizing and decalcification reaction. Said method is characterised in that it comprises an oxidation/neutralisation step which includes a step oxidizing iron into iron oxyhydroxides and/or iron oxides and a neutralizing reaction of the reaction medium.

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PCT/FR01/00390(72) Inventeurs; et
(75) Inventeurs/Déposants (pour US seulement) : TEDJAR,
Farouk [FR/FR]; 2, rue Lakanal, F-38570 Le Cheylas
(FR). JUIF, Frédéric [FR/FR]; 51 rue Victor Hugo,
F-38430 Les Crêtes-Moirans (FR).

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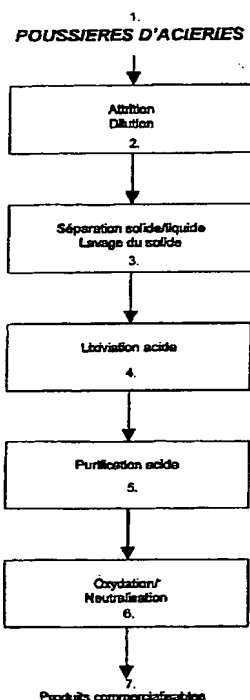
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(54) Title: METHOD FOR UPGRADING STEEL PLANT DUST

(54) Titre : PROCEDE DE VALORISATION DES POUSSIÈRES D'ACIERIES



1...STEEL PLANT DUST
2...ATTRITION DILUTION
3...SOLID/LIQUID SEPARATION WASHING SOLID
4...LEACHING ACID
5...ACID PURIFICATION
6...OXIDIZING/NEUTRALIZING
7...MARKETABLE PRODUCTS

(57) Abstract: The invention concerns a method for upgrading steel plant dust to recuperate industrially reusable compounds. Said method comprises an attrition and dilution step, a solid/liquid separating and solid washing step, an acid leaching step, and acid purification step comprising a carburizing reaction or a carburizing and decalcification reaction. Said method is characterised in that it comprises an oxidation/neutralisation step which includes a step oxidizing iron into iron oxyhydroxides and/or iron oxides and a neutralizing reaction of the reaction medium.

(57) Abrégé : L'invention a pour objet un Procédé de valorisation de poussières d'aciéries en vue de récupérer des composés réutilisables industriellement, ledit procédé comportant une étape d'attrition et de dilution, une étape de séparation solide/liquide et de lavage du solide, une étape de lixiviation acide, une étape de purification acide comprenant une réaction de cémentation ou une réaction de cémentation et de décalcification, ledit procédé étant caractérisé en ce qu'il comprend une étape d'oxydation/neutralisation, étape comprenant une réaction d'oxydation du fer en oxyhydroxydes de fer et/ou en oxydes de fer et une réaction de neutralisation du milieu réactionnel.

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Method for utilising steelworks dust

The present invention relates to a method for utilising steelworks dust.

5 The iron and steel industry, through its activity, produces waste in the form of dust.

During steel production in an electric furnace, this dust results from the following reaction.

10 Above the boiling melt, fine particles of iron are dispersed in the vapour phase and carried along by the air intake. In this air current, the particles are cooled and, under the action of the oxygen in the air, are converted into higher oxides.

15 The most volatile metals pass into the vapour phase at the operating temperature of the furnace. Under the effect of the intake, these volatile metals are oxidised and cooled by the air and end up in the form of free oxides or structures mixed with the iron oxides.

20 The composition of steelworks dust depends on the nature of the iron and steel-making method. Steelworks dust contains variable quantities of majority elements such as iron, zinc,

calcium and silicon in the form of simple or mixed oxides, and minority elements such as copper, manganese, chromium, cadmium, lead and chlorides.

5 Steelworks dust therefore contains heavy metals which are non-degradable over time and toxic to the living world.

Knowing that dust production is approximately twenty kilograms per tonne of steel, that is to say of the order of 500,000 T/year in Europe, the reprocessing of this dust constitutes a real ecological and economic undertaking.

10 Up to now, steelworks have had to place, in a Class 1 waste disposal site, their dust resulting from their activity in order for it to be stabilised. This forced movement to a waste disposal site brought about a non-negligible expenditure for the iron and steel industry.

15 The invention proposes to provide the manufacturers concerned with an economically and ecologically more advantageous alternative.

20 This is because the invention aims to eliminate this waste by using the dust from the iron and steel industry in a method enabling the different components to be separated. The purity of said components is then sufficient to enable them to constitute raw materials usable in other industries such as the mineral pigment industry among others.

25 Patent applications PCT/FR96/01202 and* PCT/FR99/00813 constitute the prior art of the invention.

Patent application PCT/FR96/01202 instances a method for preparing mineral pigments from steelworks dust. This method comprises the following steps:

- a) separation of the dust into two fractions, magnetic and non-magnetic;
- b) subjecting of the non-magnetic fraction to a basic lixiviation reaction;
- 5 c) rinsing, neutralisation and separation of the solid/liquid phases;
- d) calcination between 450°C and 650°C of the charge;
- e) treatment with sulphuric acid + catalyst of the calcined charge;
- 10 f) recovery of mineral pigments;
- g) reuse of the solutions resulting from steps c) and e) in order to precipitate other pigments.

As for patent application PCT/FR99/00813, this instances a method for treating steelworks dust with a view to the
15 recovery of utilisable elements. This method comprises:

- attrition in water;
- hydraulic classification of the charge;
- washing;
- treatment under heat;
- 20 - heat treatment between 240°C and 800°C;
- treatment with sulphuric acid with a concentration between 5% and 8%.

The methods described in the two patent applications constituting the prior art of the invention provided mineral
25 pigments or utilisable elements with a purity unacceptable for the manufacturers using this type of product.

The invention therefore aims to supply a solution by providing a method for utilising steelworks dust making it possible to obtain mineral pigments or utilisable elements of sufficient purity to make these products marketable.

5 The object of the invention is a method for utilising steelworks dust with a view to recovering industrially reusable compounds, said method having an attrition and dilution step, a step of solid/liquid separation and washing of the solid, an acid lixiviation step, an acid purification
10 step comprising a cementation reaction or a cementation and decalcification reaction, said method being characterised in that it comprises an oxidation/neutralisation step, said step comprising a reaction of oxidation of the iron into iron oxyhydroxides and/or iron oxides and a reaction of
15 neutralisation of the reaction medium carried out by means of a solution of soda, potash, sodium bicarbonate, potassium bicarbonate, ammonia or similar, in order to maintain the pH of the reaction medium substantially between 2.5 and 5.

According to one embodiment of the method, the oxidation
20 reaction is carried out by means of an oxidant taken from the group comprising hydrogen peroxide, oxygen and air.

According to one embodiment of the method, the
25 oxidation/neutralisation step is carried out substantially between 10°C and 100°C.

According to one embodiment of the method, the acid
lixiviation step is carried out by means of a sulphuric acid
solution titrating substantially between 8% and 25% acid and
30 at a temperature substantially between 50°C and 100°C.

According to one embodiment of the method, the acid purification is carried out at a pH substantially between 2 and 4 and at a temperature substantially between 20°C and 100°C.

5 According to one embodiment of the method, a hydraulic classification step is carried out after the attrition and dilution step and before the step of liquid/solid separation and washing of the solid.

10 According to one embodiment of the method, said method comprises a basic lixiviation step following the step of solid/liquid separation and washing of the solid.

15 According to one embodiment of the method, metallic zinc, zinc oxides and/or zinc ferrite are recovered from the liquid phase resulting from the oxidation/neutralisation step.

20 According to one embodiment of the method, said method comprises a step of drying and/or heat treatment of the solid phase resulting from the oxidation/neutralisation step enabling iron oxides and/or iron oxyhydroxides to be obtained.

According to one embodiment of the method, said method comprises a step of electrolysis of the liquid phase resulting from the oxidation/neutralisation step, enabling metallic zinc to be obtained.

25 According to one embodiment of the method, the solid phase resulting from the basic lixiviation step undergoes the acid lixiviation step.

30 According to one embodiment of the method, the liquid phase resulting from the basic lixiviation step undergoes a basic purification step.

According to one embodiment of the method, the basic purification step comprises a cementation reaction or a cementation and desilylation reaction.

5 According to one embodiment of the method, an electrolysis step follows the basic purification step, enabling metallic zinc and/or zinc oxides to be isolated.

Other objects and advantages of the invention will emerge in the course of the description which will be given with reference to the accompanying figures in which:

10 - Figure 1 is a schematic diagram depicting the essential steps of the method for utilising steelworks dust in their order of implementation;

- Figure 2 is a schematic diagram depicting the steps of the method according to one embodiment.

15 Figure 1 depicts schematically the essential steps of the method according to the invention, in their order of implementation.

The method for treating steelworks dust, which may contain Zn, Fe, Pb, Cd, Cr, Ca, Si and salts among other things,
20 first comprises a step of attrition of raw dust in water.

The attrition in water makes it possible to break up the dust agglomerates which have a tendency to form. This step facilitates the subsequent separation of the charge into two fractions:

25 - dust of size greater than 40 microns;

- dust of size smaller than 40 microns.

This moist phase treatment step assumes great importance in the method and therefore constitutes, as such, an essential step as it makes it possible to:

- speed up the dissolving of the soluble salts;
- 5 - liberate the metallic fractions masked by the oxides adsorbed on the surface.

The charge thus obtained is next diluted in water. This dilution serves to wholly or partially solubilise the salts, such as chlorides and/or sulphates.

- 10 A step of separating the solid/liquid phases generated is then carried out, which may be accompanied by washing of the solid phase. The aim of this washing is to eliminate as much as possible the salts still present in the solid phase where they were not solubilised in the attrition and
- 15 dilution step. Finally, the solid phase is separated from the wash water.

This salt elimination step is important as it conditions, in part, the degree of purity of the elements or compounds which will be isolated subsequently.

- 20 The solid phase may next undergo an acid lixiviation step. For this, the solid is treated with an acid solution, for example a sulphuric acid solution, in order to totally or partially dissolve the metals such as iron, zinc, lead, cadmium, nickel and chromium. The liquid phase, containing
- 25 dissolved metals, is separated from the solid phase.

The silicon, which is insoluble, is contained in the solid phase. The acid lixiviation step has therefore made it possible, among other things, to separate the silicon from the other elements or compounds to be utilised.

The liquid phase, which is acid, is next purified in order to totally or partially eliminate the metals such as lead, cadmium, nickel and chromium as well as the calcium, if necessary.

5 This acid purification step consists in adding, to the acid liquid phase, with a pH situated between 2 and 4, sodium fluoride in order to precipitate the calcium if necessary, and powdered zinc and/or iron in order to cement the lead, cadmium, nickel and chromium.

10 The powdered zinc used in this purification step can be that obtained by the method for the invention.

Cementation means deposition by reduction. During cementation, a noble body, oxidised and dissolved, is reduced by another metallic body added to the solution in powdered form.

The liquid phase, resulting from the acid purification step, is then recovered. It comprises the metals iron and zinc.

A step, including an oxidation reaction and a neutralisation reaction, said reactions capable of taking place simultaneously, is carried out on the liquid phase comprising iron in particular. The aim of this step is to precipitate the iron in the form of iron oxyhydroxides and/or iron oxides, while the zinc remains in solution in the reaction medium.

25 The oxidation reaction is carried out by means of a liquid oxidant such as hydrogen peroxide or a gaseous oxidant such as air or oxygen at a pH maintained between 2.5 and 5 by means of a basic solution such as a soda solution or similar. This pH condition is important in order to avoid the precipitation of the zinc at the same time as that of the iron.

At the end of this oxidation/neutralisation step, the following are obtained:

- directly, marketable products such as iron oxyhydroxides and/or iron oxides; and
- 5 - indirectly, marketable products such as zinc oxides, zinc ferrites and metallic zinc, after treatment of the reaction medium.

Figure 2 depicts a method for utilising steelworks dust according to one embodiment of the invention.

- 10 The description of the method shown schematically in Figure 2 which follows will be accompanied by numerical values by way of example. These numerical values are in no way limitative.

- 15 It will be considered for the remainder of the description that one tonne of steelworks dust undergoes the utilisation method described below.

The first step of the method for utilising steelworks dust is an attrition step. This moist phase treatment step is carried out in an attrition unit in the presence of water.

- 20 The ratio $R1 = (\text{dry matter of steelworks dust in kg}) / (\text{volume of water in litres})$ must be chosen so as to allow a suitable friction between the solid particles while providing a sufficient volume of liquid to enable dissolving of the water-soluble fractions such as salts.

- 25 The ratio $R1$ is substantially between 2.2 and 2.7. This step is carried out for 10 to 20 minutes.

The slurry, constituted by the attrited charge of steelworks dust mixed in the water, is next diluted in water in a manner sufficient to:

- complete the dissolving of the salts, in particular the chlorides and sulphates;
- put the very fine particles into suspension; and
- provide a density adapted to hydraulic classification.

5 The diluting of the slurry makes it possible to achieve a dry mass concentration substantially between 4% and 15%.

The slurry thus diluted next undergoes a hydraulic classification step. The hydraulic classification is advantageously carried out in cut-point hydrocyclones adapted to the granulometry of the dust introduced therein.

10

The slurry can pass through a hydrocyclone having a cut-off between 30 and 100 microns. The differential pressure is substantially between 0.6 bar and 1 bar and the ratio $R2 = (\text{incoming slurry flow rate})/(\text{water counterflow rate})$ is substantially between 100 and 200.

15

At the end of the cycloning, the following are distinguished:

- the underflow rich in magnetite and possibly comprising carbon and iron; and

20

- the overflow rich in heavy metals, in the upper part of the vertical cylinder of the hydrocyclone.

The underflow enables recovery of 100 kg to 200 kg of dry matter while the overflow enables recovery of 800 kg to 900 kg of dry matter starting with one tonne of treated steelworks dust.

25

The underflow charge can form the subject for example of a recharging of the furnace after decantation, spinning and

drying or can form the subject of a utilisation of the magnetite fraction after extraction.

The diluted slurry, or the overflow in the case where the diluted slurry has undergone a hydraulic classification, next forms the subject of a solid/liquid separation step. This separation can be performed, for example, by filtration by means of a filter press. The solid phase is then recovered on the filter. Simultaneously, the solid can be washed in order to eliminate the salts which were not dissolved in the dilution water of the previous attrition and dilution step.

At the end of this solid/liquid separation and washing step, the chloride and sulphate concentration in the solid is less than around 1000 ppm for each of the two salts.

The solid, obtained at the end of the solid/liquid separation and washing step, can next undergo a basic lixiviation step. This step makes it possible to solubilise the free metals such as zinc, lead, cadmium, nickel and chromium, as well as silicon. On the other hand, iron is not solubilised.

Depending on the origin of the dust, between 10% and 50% by weight of solid subjected to the basic lixiviation step is solubilised.

The bases which can be used are chosen from the group comprising: soda, potash and sodium bicarbonate. Other bases can advantageously be used.

In the case of soda, a concentration substantially between 240 g/l and 450 g/l is required.

The ratio $R3 = (\text{dry matter in kg}) / (\text{reaction volume in litres})$ can be between 0.1 and 0.3.

The basic lixiviation reaction is carried out at a temperature substantially between 70°C and 120°C. The duration of the step is a function of the quantity of compounds to be solubilised.

5 At the end of this step, a filtration of the solution enables separation of the solid and liquid phases. The solid phase, rich in iron, is directed to an acid lixiviation step while the liquid phase, rich in metals other than iron and possibly comprising silicon, can be
10 directed to a basic purification step which will be described a little later in the description.

Generally speaking, an acid lixiviation makes it possible to solubilise all metals such as iron, zinc, lead and cadmium and to make silicon insoluble.

15 Between 80% and 95% by weight of solid subjected to the acid lixiviation step is solubilised.

Where the basic lixiviation step is bypassed, the solid resulting from the solid/liquid separation and washing step undergoes an acid lixiviation step which leads to
20 solubilisation of the iron, zinc, lead, nickel, chromium and cadmium it might contain, while the silicon remains insoluble.

Where the basic lixiviation step takes place, the iron contained in the solid phase obtained at the end of the
25 basic lixiviation step is solubilised, as is the zinc tied up in ferrite form.

The acid used for the acid lixiviation step is, for example, sulphuric acid titrating substantially between 8% and 25% acid. This acid lixiviation step is carried out at a
30 temperature substantially between 50°C and 100°C. The

duration of the step depends on the quantity of compounds to be solubilised.

The ratio $R4 = (\text{dry matter in kg})/(\text{reaction volume in litres})$ is substantially between 0.05 and 0.15.

- 5 At the end of the acid lixiviation step, the reaction medium is, for example, filtered on a filter press so as to separate the solid phase containing the insoluble compounds such as for example silicon, and the acid aqueous phase or filtrate containing iron and which may also contain the
10 solubilised elements such as zinc, lead, cadmium, nickel and chromium.

The iron and zinc concentrations are substantially between 30 g/l and 60 g/l for the iron and between 5 g/l and 20 g/l for the zinc.

- 15 An acid purification step follows the acid lixiviation step. This purification step comprises a cementation reaction and possibly a decalcification reaction.

- The filtrate from the acid lixiviation step is purified by cementation of certain metals such as lead, cadmium, nickel
20 and chromium by the addition of zinc and/or iron. The quantity of zinc and/or iron to be added is a function of the quantity of compounds to be cemented.

For one tonne of initial steelworks dust, up to around 200 kg of zinc are necessary.

- 25 Cementation takes place at a pH substantially between 2 and 4 and at a temperature substantially between 20°C and 100°C.

Decalcification can also be carried out, if necessary, during this acid purification step, simultaneously or not with the cementation reaction. In order to decalcify the

filtrate from the acid lixiviation reaction, sodium fluoride is added to said filtrate in order to precipitate the calcium.

5 At the end of this acid purification step, the reaction medium is, for example, filtered on a filter press so as to separate the solid and liquid phases.

10 The filtrate resulting from the acid purification step has a total concentration of lead, cadmium, nickel, chromium and calcium substantially less than 5 mg/l and on the other hand contains a significant concentration of iron and zinc.

15 The solid phase resulting from the acid purification step can be neutralised and washed so as to achieve a pH substantially between 6.5 and 7. This solid phase can be utilised subsequently as it contains heavy metals such as lead, zinc, etc in sufficient quantity.

The step following the acid purification step is the so-called oxidation/neutralisation step. The aim of this step is to precipitate the iron in the form of iron oxyhydroxides and/or iron oxides following its oxidation.

20 To carry out the oxidation reaction, use can be made of a liquid oxidant of hydrogen peroxide type at a concentration substantially between 5% and 35% or a gaseous oxidant of air or oxygen type.

25 It is imperative that this oxidation/neutralisation step be carried out at a pH substantially between 2.5 and 5. Adjustment of the pH into this pH range is made simultaneously with the oxidation reaction by the addition of a base such as soda at a concentration substantially between 100 g/l and 200 g/l. The reaction is carried out at
30 a temperature substantially between 10°C and 100°C.

With the oxidation/neutralisation reaction complete, the reaction medium is, for example, filtered on a filter press enabling separation of the solid phase containing iron oxyhydroxides and/or iron oxides and the liquid phase or
5 filtrate containing zinc at a concentration which may reach 50 g/l.

The solid phase recovered on the filter is washed and dried at a temperature substantially between 80°C and 150°C, possibly followed by a heat treatment carried out at a
10 temperature substantially between 400°C and 850°C. The iron oxides and iron oxyhydroxides obtained after drying and/or heat treatment constitute products marketable in the pigment field.

The filtrate resulting from the oxidation/neutralisation
15 step contains essentially zinc. In order to utilise this zinc, three ways can be envisaged:

- electrolysis of the filtrate enabling isolation of metallic zinc;
- synthesis of zinc ferrite;
- 20 - obtaining of zinc oxides.

All these products constitute marketable products.

As mentioned above, the basic liquid phase resulting from the basic lixiviation step can undergo a basic purification step. This purification comprises a cementation reaction,
25 perhaps even a desilylation reaction, if necessary.

The basic liquid phase, rich in metals other than iron, is cleared of certain metals such as lead, cadmium, nickel and chromium by the addition of zinc in sufficient quantity to cement all the metals mentioned above.

This cementation reaction is carried out at a temperature substantially between 20°C and 100°C.

The desilylation reaction enables precipitation of the silicon. This reaction can be carried out simultaneously with the cementation reaction by adding slaked lime to the medium.

For example, in a particular composition, for one tonne of treated steelworks dust, up to 9 kg of slaked lime is necessary.

At the end of this basic purification step, the reaction medium is, for example, filtered on a filter press so as to separate the basic solid phase and the basic liquid phase or basic filtrate.

The basic solid phase can contain metals such as lead, cadmium, nickel and chromium. It can also contain silicon. The basic solid phase is neutralised and washed. It can form the subject of a utilisation of the metals it contains by subsequent treatments.

The basic filtrate, zinc-enriched, can undergo electrolysis. The product, collected after electrolysis and with an acceptable faradaic efficiency, comes in the form of powdered zinc which, once washed and dried, makes it possible to obtain powdered metallic zinc and/or zinc oxides, which are marketable products.

The electrolysed basic filtrate, zinc-depleted, can be reused in the basic lixiviation step.

By way of example, without it being exhaustive, starting with one tonne of steelworks dust as the initial substance, the following are obtained:

- 600 kg of iron oxyhydroxides and/or iron oxides at the end of the drying and/or the heat treatment;
- 300 kg of co-products.

The advantages of this method are:

- 5 - being global since it aims to utilise all metals liable to be present in steelworks dust;
- being economically and ecologically advantageous compared with placing this type of waste in a Class 1 waste disposal site;
- 10 - providing a better purity of the products thus utilised by the method compared with the methods of the prior art;
- making these utilised products marketable.

CLAIMS

1. A method for utilising steelworks dust with a view to recovering industrially reusable compounds, said method having an attrition and dilution step, a step of
5 solid/liquid separation and washing of the solid, an acid lixiviation step, an acid purification step comprising a cementation reaction or a cementation and decalcification reaction, said method being characterised in that it comprises an oxidation/neutralisation step, said step
10 comprising a reaction of oxidation of the iron into iron oxyhydroxides and/or iron oxides and a reaction of neutralisation of the reaction medium carried out by means of a solution of soda, potash, sodium bicarbonate, potassium bicarbonate, ammonia or similar, in order to maintain the pH
15 of the reaction medium substantially between 2.5 and 5.
2. A method according to Claim 1, characterised in that the oxidation reaction is carried out by means of an oxidant taken from the group comprising hydrogen peroxide, oxygen and air.
- 20 3. A method according to one of the preceding claims, characterised in that the oxidation and neutralisation step is carried out substantially between 10°C and 100°C.
4. A method according to one of the preceding claims, characterised in that the acid lixiviation step is carried
25 out by means of a sulphuric acid solution titrating substantially between 8% and 25% acid and at a temperature substantially between 50°C and 100°C.
5. A method according to one of the preceding claims, characterised in that the acid purification is carried out
30 at a pH substantially between 2 and 4 and at a temperature substantially between 20°C and 100°C.

6. A method according to one of the preceding claims, characterised in that a hydraulic classification step is carried out after the attrition and dilution step and before the step of liquid/solid separation and washing of the solid.

7. A method according to one of the preceding claims, characterised in that it comprises a basic lixiviation step following the step of solid/liquid separation and washing of the solid.

8. A method according to one of the preceding claims, characterised in that metallic zinc, zinc oxides and/or zinc ferrite are recovered from the liquid phase resulting from the oxidation/neutralisation step.

9. A method according to one of the preceding claims, characterised in that it comprises a step of drying and/or heat treatment of the solid phase resulting from the oxidation/neutralisation step enabling iron oxides and/or iron oxyhydroxides to be obtained.

10. A method according to one of the preceding claims, characterised in that it comprises a step of electrolysis of the liquid phase resulting from the oxidation/neutralisation step, enabling metallic zinc to be obtained.

11. A method according to Claim 7, characterised in that the solid phase resulting from the basic lixiviation step undergoes the acid lixiviation step.

12. A method according to Claim 7, characterised in that the liquid phase resulting from the basic lixiviation step undergoes a basic purification step.

13. A method according to Claim 12, characterised in that the basic purification step comprises a cementation reaction or a cementation and desilylation reaction.

5 14. A method according to Claim 12 or 13, characterised in that an electrolysis step follows the basic purification step, enabling metallic zinc and/or zinc oxides to be isolated.

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STEELWORKS DUST

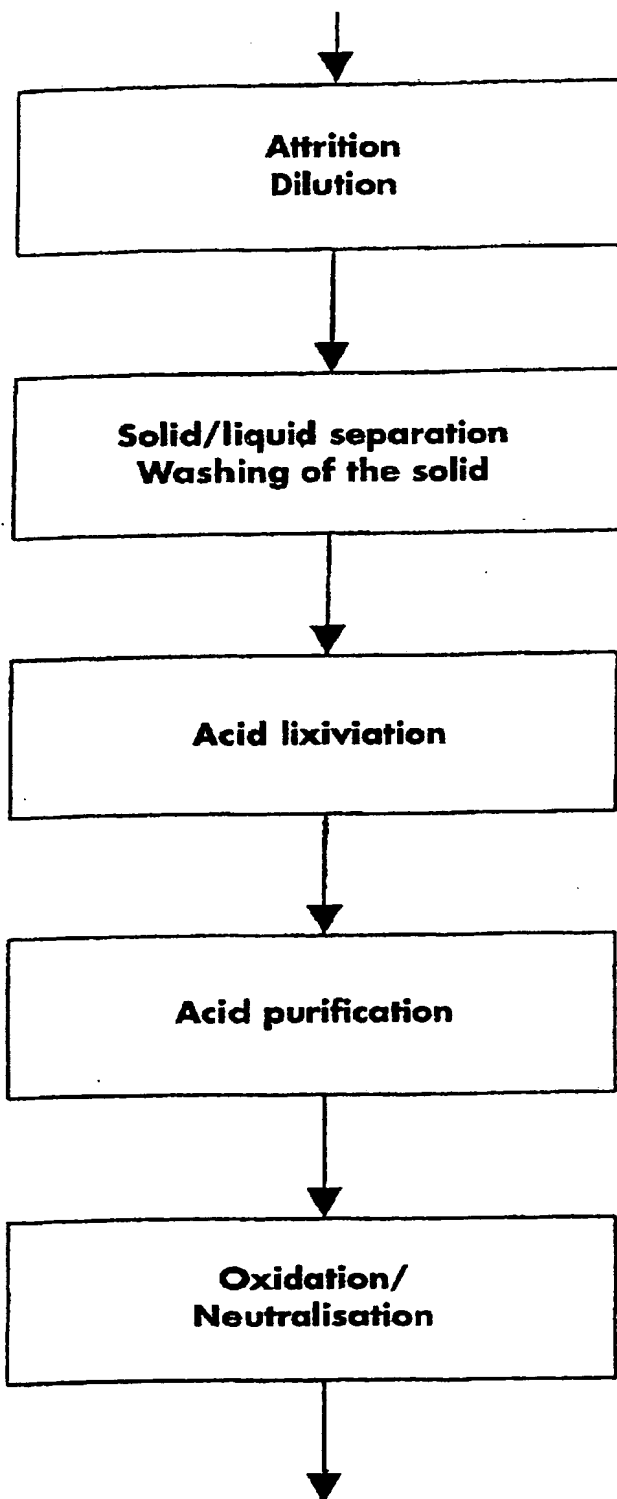


Fig. 1

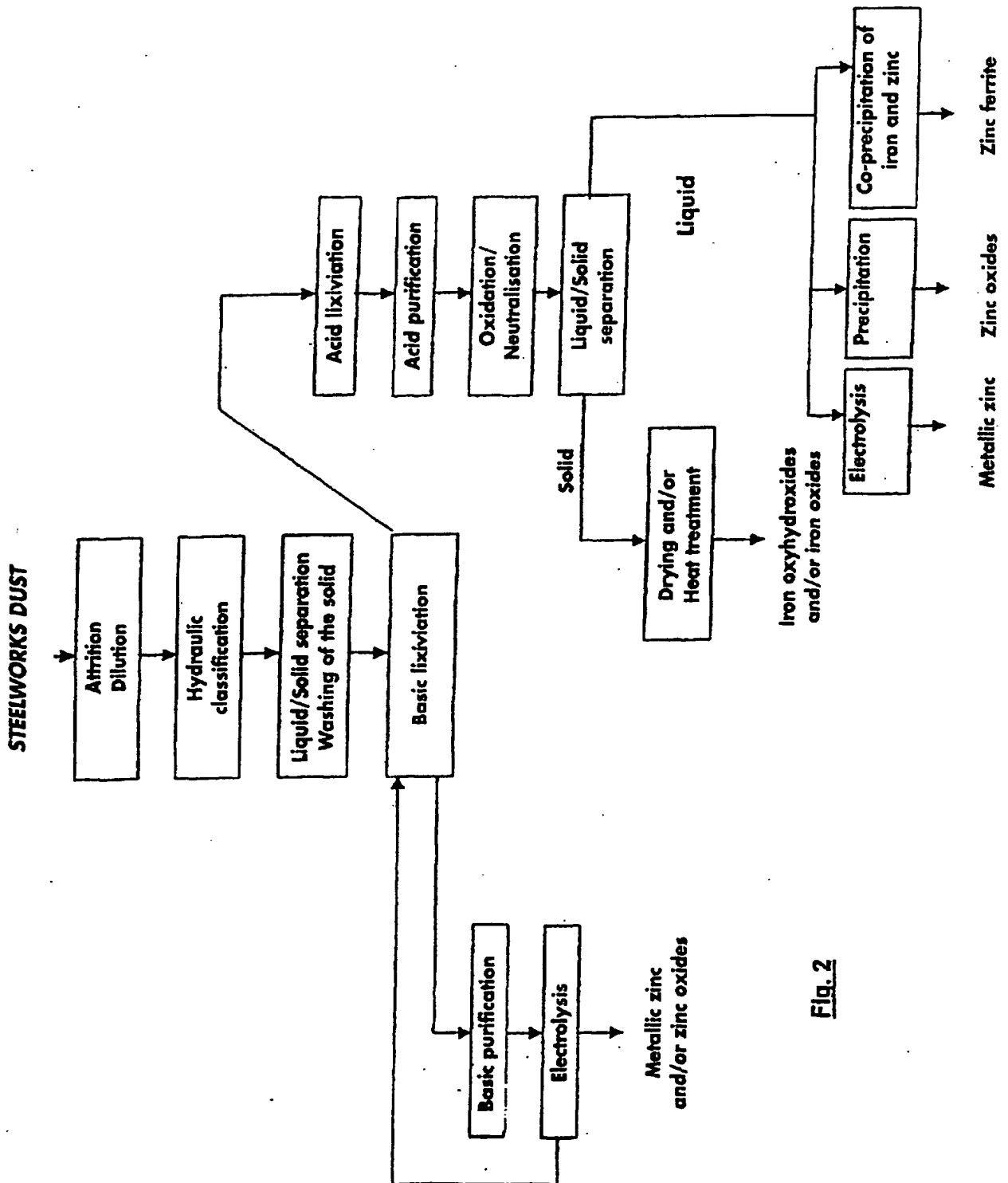


Fig. 2